

AD-A284 451



REAL-TIME MONITOR AND CONTROL OF MBE GROWTH OF HgCdTe  
BY  
SPECTROSCOPIC ELLIPSOMETRY

MONTHLY PROGRESS  
TECHNICAL REPORT NO.4  
Reporting Period: Sept.30, 1993 - Oct.30, 1993

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DEPARTMENT OF THE ARMY  
U.S. ARMY COMMUNICATION-ELECTRONICS COMMAND  
FORT MONMOUTH, NJ 07703-5000

CONTRACT #DAAB07-93-C-U008

J. A. WOOLLAM CO., INC  
650 J STREET, SUITE 39  
LINCOLN, NE 68508

Effective date of contract: May 19, 1993  
Contract Expiration Date: May 18, 1995

Distribution Statement A: Approved for public release; distribution is unlimited.

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Contract #DAAB07-93-C-U008  
"Real-Time Monitor and Control of MBE Growth of HgCdTe  
by Spectroscopic Ellipsometry"

**Summary of Previous Related Work  
& Proposed Tasks for this Contract  
11/5/93**

*Overall Goal of this Contract: Compositional control of  $\text{Hg}_{1-x}\text{Cd}_x\text{Te}$  (MCT) during MBE growth, using a commercially viable spectroscopic ellipsometer system. The run-to-run composition reproducibility, as well as the composition uniformity during growth, should be controlled to within  $\pm 0.001$ .*

*Main Task of this Contract: Develop a knowledge base which includes a quantitative understanding of MCT growth by MBE from an optical/ellipsometric standpoint, and practical application of this understanding to effect real-time composition control. The knowledge base, which will be developed through extensive and systematic in-situ ellipsometry experiments, will be comprehensive and general enough to facilitate the transfer of this composition control technology to other MCT crystal growers.*

## **Previous Work**

- **Developed a prototype 44 wavelength spectroscopic (or 'multi- $\lambda$ ') ellipsometer system**

This system, both hardware and software, is specifically designed for acquiring and analyzing real-time in-situ ellipsometric data during material deposition and/or processing. The development of this system was partially supported by the Phase I work of this contract. Funding from other government research contracts, in addition to internal funding from the J.A. Woollam Co., also supported the development of this instrument. We are currently in the process of commercializing this instrument; preliminary versions have been already been delivered to 2 customers.

- **In-situ experiments with the prototype multi- $\lambda$  ellipsometer:**

- Rensselaer Polytechnic Institute (RPI) - MCT growth by MOVPE
- Arizona State University (ASU) -  $\text{Al}_x\text{Ga}_{1-x}\text{As}$  growth by MBE
- University of Nebraska at Lincoln -  $\text{SiO}_x\text{N}_y$  growth and semiconductor etching by ECR
- C VI Laser Corp, Albuquerque, NM - optical coatings deposition by e-beam evaporation

As part of other government research contracts, we have tested the prototype instrument in a number of in-situ environments. Each environment has its own challenges, in terms of how to measure accurate in-situ ellipsometric data in the growth chamber, and how to analyze the raw ellipsometric data to obtain film thickness, composition, temperature, etc. (optical constants and growth mechanisms for each material system are dramatically different). We have demonstrated some exciting feedback-controlled growth results at ASU (sub-monolayer thickness control of GaAs/AlAs layers) and RPI (CdTe growth rate control and MCT

composition control). However, these preliminary results came from specific experiments designed to demonstrate the concept of feedback-controlled growth by ellipsometry; we do not have enough experience or the optical constant library necessary to perform controlled growth of arbitrary structures or materials. On-going work is focusing on: obtaining more extensive optical constant libraries, repeating the controlled growth experiments, and verifying the results with other analysis techniques.

## Tasks to be Performed under this Contract

- **Install and test a multi- $\lambda$  in-situ ellipsometer on the NVESD MCT MBE chamber**

The chamber is currently being upgraded to provide ellipsometry ports and more stable substrate rotation. Adequate angular stability of the substrate rotation stage, which is necessary for accurate ellipsometric data acquisition during growth, must be verified.

- **Develop a library of temp. & composition dependent optical constants for  $\text{Hg}_{1-x}\text{Cd}_x\text{Te}$**

Ellipsometry is an optical technique; accurate material optical constants must be measured *before* composition can be extracted from the raw ellipsometric data. The required optical constants are *not* available in the literature, nor will they be trivial to measure. It is important that they be measured under actual growth conditions, i.e., MCT optical constants obtained during MOVPE growth may not be suitable for MCT growth by MBE, due to differences in substrate temperature, growth mechanism, and surface morphology. Within the next couple of months, a very systematic and controlled set of experiments will be devised to facilitate the measurement of temperature and composition dependent MCT optical constants.

- **Quantitatively study the MCT MBE growth process**

Using the optical constant library described above, raw ellipsometric data must be analyzed using a model-dependent regression analysis to extract physical parameters such as thickness, composition, temperature, and surface roughness during growth. MCT growth by MBE is a very complicated process; it requires precise temperature control, and the surface morphology and composition can change dramatically during deposition. Ellipsometry is highly sensitive to all of these effects. It is therefore extremely important that we are able to precisely model the ellipsometric data to unambiguously separate out these effects. For example, a  $10^\circ$  change in temperature should not be interpreted as a 0.01 change in composition. This task is very computationally intensive; fortunately, most of the software required for this data analysis has already been developed by the J.A. Woollam Co.

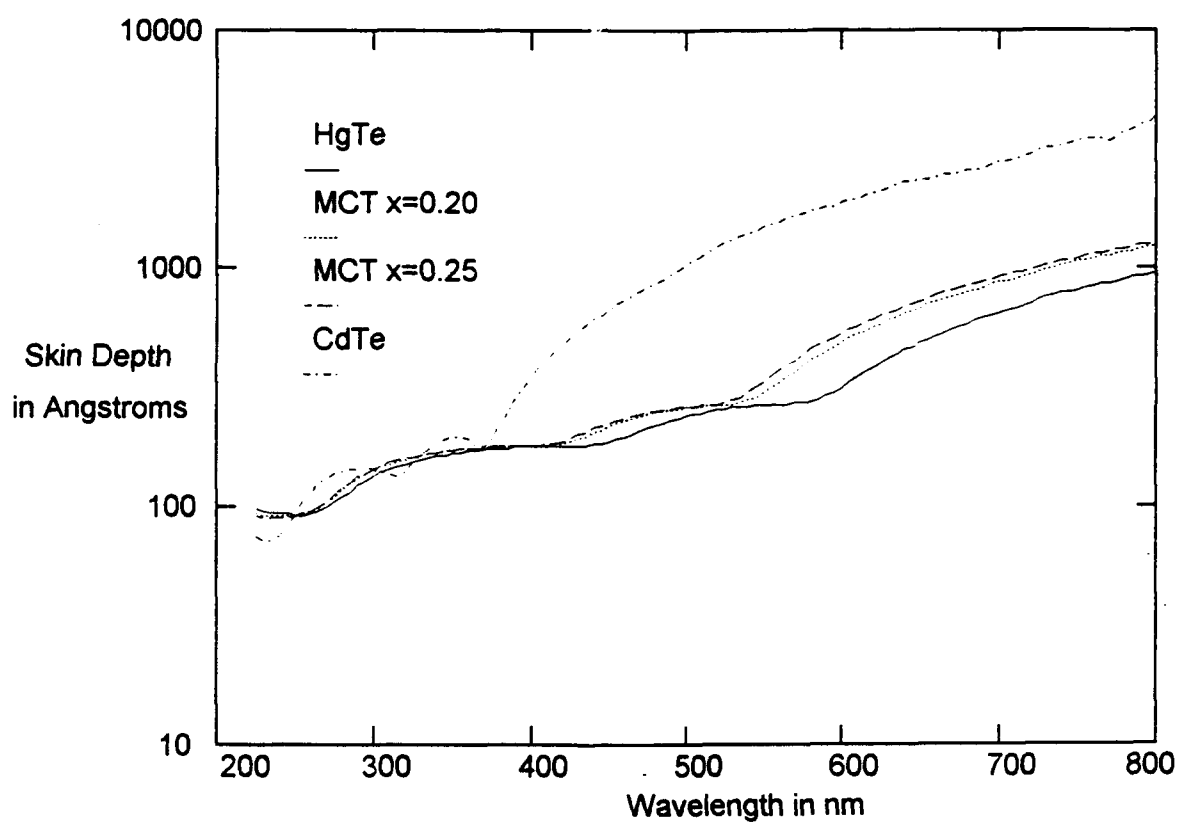
- **Effect feedback control of the MCT composition**

Using the models developed in the previous task, the multi- $\lambda$  ellipsometer system will accurately measure the MCT surface composition, in real-time, during growth. Customized software will be written (in Visual Basic) to interface the multi- $\lambda$  in-situ ellipsometer system with the MBE machine. By adjusting the Cd cell temperature, closed-loop feedback control of composition will be effected. Many controlled-growth depositions will be performed; the resulting samples will be tested by other analysis techniques to verify the control accuracy and long-term reproducibility.

Available Codes	
Dist	Special
A-1	

# Penetration Depth of Light in $\text{Cd}_x\text{Hg}_{1-x}\text{Te}$

(using room temp. optical constants; Skin Depth =  $\lambda/4\pi k$ )



**REAL-TIME MONITOR AND CONTROL OF MBE GROWTH OF HgCdTe  
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**MONTHLY PROGRESS  
TECHNICAL REPORT NO.5  
Reporting Period: Oct..30, 1993 - Nov..30, 1993**

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FORT MONMOUTH, NJ 07703-5000**

**CONTRACT #DAAB07-93-C-U008**

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Reporting Date: Dec. 13, 1993

All of the machine parts for your multi- $\lambda$  ellipsometer system have been received from the shop; we are in the process of having them anodized. The electronics board and control box are currently being assembled. We should have the complete system assembled within the next two weeks. We normally test the systems in-house for 1-2 weeks before shipping to make sure that everything is working properly. This implies a tentative shipping date in the 3rd week of January. I would propose the following: at the time of next month's report (which you will receive in the 1st week of January), we can evaluate the status of the system and set the final delivery date for the system. This would give us a couple of weeks to make travel arrangements, etc. I don't think that it will be necessary for you to come to Lincoln for training, as we should be able to cover all the necessary details in your lab (we even have a preliminary hardware manual written for the system).

Preliminary testing has indicated that the two potential hardware modifications that I mentioned in the last report, a new lamp housing design and different alignment detector location, work quite well. The combination of the two modifications can boost the signal throughput by almost an order of magnitude. We plan on integrating the different alignment detector location modification into your system (which improves the alignment reproducibility and provides a 2-3x improvement in signal intensity). We may not have the improved lamp housing ready for shipping by mid-January; however, we can still ship the previous lamp housing and exchange it with the new lamp housing at a later date.

On the software front, I have finished the 'multi-sample' analysis capability for dynamic data. This powerful feature will allow us to simultaneously extract optical constants and surface morphology effects (such as surface roughness and/or interdiffusion) from the insitu ellipsometric data. Unfortunately, I have not had time to write up any further data analysis from our previous trips, but I do believe that this latest software addition is flexible enough to handle any dynamic data analysis that we might encounter. I do not plan on doing any further software development to support the multi- $\lambda$  system (except for the 'custom' interface to your MBE system).

Submitted by



Blaine Johs

Principle Investigator

**REAL-TIME MONITOR AND CONTROL OF MBE GROWTH OF HgCdTe  
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**MONTHLY PROGRESS  
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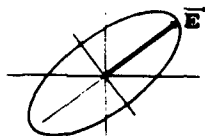
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## J. A. WOOLLAM CO., INC.

—Research & Instrumentation—

Reporting Date: Feb 7, 1994

This month we shipped a 44 wavelength insitu ellipsometer system to the NVESD. I have also devised a proposed measurement method to determine MCT optical constants as a function of temperature and composition. This experiment would probably be one of the first insitu ellipsometer measurements performed after the new MBE chamber is operational.

We have also begun to decipher some of the insitu data we acquired at RPI during MOVPE growth of MCT. For example, we initially thought that the ellipsometric data was indicating a high degree of interdiffusion between CdTe and HgTe layers. However, our current analysis provides an alternate, and hopefully more believable, explanation: even though the  $H_2$  flow through the Hg boat is shut off during CdTe growth, the vapor pressure of the Hg is high enough that Hg still gets incorporated into the growing 'CdTe' buffer layer. Instead of growing CdTe, MCT is grown. In fact, when switching from HgTe to CdTe growth modes, a layer of compositionally graded MCT is grown. We are using this graded layer (which 'accidentally' exists; an abrupt interface was desired) to extract growth temperature optical constants for MCT at a range of compositions.

Submitted by

Blaine Johs

Principle Investigator

### **Proposed Measurement Method:**

#### **Insitu Optical Constants Library for Ternary Semiconductors as a function of Temperature and Composition**

**Ideal Objective:** Determine exact, intrinsic, optical constants for the ternary semiconductor system of interest for the complete range of temperatures and compositions, by performing insitu ellipsometric measurements. Absolutely correct values for temperature and composition (determined by other measurement techniques, such as thermocouple, pyrometry, PL, X-ray diffraction, etc.) should be associated with each measured optical constant spectra. *This objective is not achievable in a reasonable time frame (would require a separate growth run and exsitu sample analysis for each composition) with currently available deposition systems (inadequate wafer positioning reproducibility) and measurement tools (inadequate insitu temperature measurement accuracy).*

**Realistic Objective:** Determine (as accurately as possible) optical constants for the ternary semiconductor system of interest, for a few temperatures in the typical growth regime of the system, and at a limited number of representative compositions. Initially, only nominal values for temperature and composition will be assigned to the determined optical constant spectra. These nominal values may not be accurate in an absolute sense, but they will still enable the reproducible insitu determination of temperature and composition in a relative manner. The nominal values for temperature and composition assigned to each optical constant spectra can be replaced with more accurate values when that information becomes available (through extensive exsitu analysis of subsequently grown samples). For example, using a measured optical constant library with nominal values, one might reproducibly grow layers at 600°C (nominal) of composition 0.25 (also nominal). Two months later, after growing 20 of these layers at the exact same nominal temperature and composition, a very accurate pyrometer measures the actual wafer temperature to be 613°C, and PL measurements indicate that the actual composition is 0.268. These accurate absolute measurements do not invalidate the previously measured optical constant library; only the nominal temperature and composition values need to be replaced to improve the absolute accuracy of the optical constant library. The bottom line is that reproducibility (or relative accuracy) is more important and more readily achievable than absolute accuracy (which can still be developed over time). *It should be possible to realize this objective by following the procedure described in this proposal.*

**Procedure:**

(to measure optical constants at nominal temperatures  $t_x = t_1 \dots t_n$  and compositions  $c_y = c_1 \dots c_m$ )

1. Ramp wafer to blow-off oxide, and grow a thick buffer layer (which should be a binary semiconductor) to provide a smooth, high-quality surface. During the buffer layer growth, the wafer temperature should be stabilized and maintained at  $t_1$ .
2. Grow a layer (300-500Å) of composition  $c_y$ , with the temperature stabilized at  $t_x$ . As the optical constants for  $c_y$  are different from the optical constants of the buffer layer, interference effects will be observed in the ellipsometric data. By performing a sophisticated multi-model analysis on this data, highly accurate optical constants of the growing layer can be determined, even when the layer is grown on top of a complex multi-layer structure. In addition, the growth rate that is also extracted from the analysis may be used to estimate the composition of the layer. Because this analysis approach uses multiple instances of ellipsometric data acquired during the layer growth (as opposed to performing a single ellipsometric measurement of the material surface after the growth), the optical constants are 'over determined'. This minimizes inaccuracies in the determined optical constants due to ellipsometer measurement errors (no ellipsometer is perfectly accurate, especially when performing insitu measurements through 'strain-free' windows) and potential material interface and surface non-idealities.
3. Grow a 300-500Å buffer layer with the temperature remaining stabilized at  $t_x$ . There are a number of reasons for growing this buffer layer. First of all, during the growth of the buffer layer, the effusion cell temperature of the composition determining element can be ramped and stabilized to the temperature required to grow the next composition. Secondly, optical constants can be extracted from the buffer layer growth (using multi-model analysis). If the material quality remains good and the temperature stays at  $t_x$ , the optical constants for each buffer layer should be identical. This provides a good self-consistency check of the temperature stability and determined optical constant accuracy. And lastly, the buffer layer provides 'optical contrast' to ensure that the growth of the next composition generates interference oscillations in the ellipsometric data.
4. Repeat steps 2 and 3 for the number of compositions desired.
5. Repeat steps 2-4 for the number of temperatures desired

**Proposed Growth Structure**  
(to determine optical constants at 2 temperatures and 3 compositions)

$\approx 400\text{\AA}$ buffer layer at temperature $t_2$
$\approx 400\text{\AA}$ layer, composition $c_3$ , temp. $t_2$
$\approx 400\text{\AA}$ buffer layer at temperature $t_2$
$\approx 400\text{\AA}$ layer, composition $c_2$ , temp. $t_2$
$\approx 400\text{\AA}$ buffer layer at temperature $t_2$
$\approx 400\text{\AA}$ layer, composition $c_1$ , temp. $t_2$
$\approx 400\text{\AA}$ buffer layer at temperature $t_2$
$\approx 400\text{\AA}$ layer, composition $c_3$ , temp. $t_1$
$\approx 400\text{\AA}$ buffer layer at temperature $t_1$
$\approx 400\text{\AA}$ layer, composition $c_2$ , temp. $t_1$
$\approx 400\text{\AA}$ buffer layer at temperature $t_1$
$\approx 400\text{\AA}$ layer, composition $c_1$ , temp. $t_1$
$\approx 1\text{ }\mu\text{m}$ buffer layer at temperature $t_1$
Substrate

**Advantages of this Method:**

1. Can be used with almost any lattice matched ternary semiconductor system, independent of the growth technique (MBE, MOVPE, etc.).
2. The entire library of optical constants is determined on the same wafer and in the same experiment. This eliminates errors in wafer angle of incidence reproducibility, and minimizes errors in temperature reproducibility.
3. The procedure is simple enough (a single 1-3 hour growth run) that it could easily be performed on different growth chambers to account for small differences in window effects, material quality, growth methods, etc. At this point, there is no guarantee that the measured optical constant library would be universally applicable for insitu growth control on other chambers, although this would certainly be a desirable goal.
4. Determining the optical constants of the material during growth is far more accurate (and useful) than measuring the optical constants of a static surface.

**Other Important Notes on Temperature Dependence of Optical Constants**

1. In general, the temperature dependence of material optical constants is much smaller than the composition dependence. Therefore, if the material is grown only within a small range of temperatures, it may be adequate to determine compositionally dependent optical constants only at a single typical growth temperature.
2. It may be desirable to measure temperature dependent optical constants for the substrate from room temperature to growth temperature. This would enable the ellipsometer to measure the temperature of the wafer during heating, and determine the substrate temperature before deposition. To determine temperature dependent optical constants for the substrate, simply acquire ellipsometric data while varying the substrate temperature (after the oxide has been blown-off and a buffer layer has been grown).

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